

Development of One Meter Long Double-Sided CeO_2 Buffered Ni-5at.%W Templates by Reel-to-Reel Chemical Solution Deposition Route

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Abstract—High performance long-length coated conductors fabricated using various techniques have attracted a lot of interest recently. In this work, a reel-to-reel design for depositing double-sided coatings on long-length flexible metallic tapes via a chemical solution method is proposed and realized. The major achievement of the design is to combine the dip coating and drying processes in order to overcome the technical difficulties of dealing with the wet films on both sides of the tape. We report the successful application of the design to fabricate a one-meter-long double side coated $\text{CeO}_2/\text{Ni} - 5\text{at}\%\text{W}$ template. The CeO_2 films on both sides exhibit a dense, crack-free morphology, and a high fraction of cube texture on the surface. Homogeneity studies on global texture over the length also reveal that the average full width at half maximum values of the in-plane and out-of-plane orientation on the CeO_2 layer are 7.2° and 5.8° with standard deviation of 0.26° and 0.34° , respectively, being indicative of the high quality epitaxial growth of the films prepared in the continuous manner. An all chemical solution derived $\text{YBCO}^{\text{Low-TFA}}/\text{Ce}_{0.9}\text{La}_{0.1}\text{O}_2/\text{Gd}_2\text{Zr}_2\text{O}_7/\text{CeO}_2$ structure is obtained on a short sample, demonstrating the possibility of producing long-length texture templates for coated conductors by this low cost deposition route.

I. INTRODUCTION

OVER the years, several milestones have been achieved on the development of the second generation (2G) coated conductors (CCs) based on $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) superconducting layers, as evidenced by industrialization of long-length 2G wires fabricated worldwide by various routes [1]–[4]. In order to fulfill the requirements for large-scale commercial applications, several issues in CCs fabrication have to be taken into consideration, such as further improving performance, reducing costs, increasing production rate, etc. During the last

few years, it has been demonstrated that rolling assisted biaxially textured substrates (RABiTS) combined with chemical solution deposition (CSD) is a very promising method for cost efficient fabricating 2G wires.

The CSD method has many advantages of over other thin film deposition techniques [5]. Besides lower capital investments and the ability of scaling up easily and control of the composition at atomic scale, this method allows to simultaneously coat a double-sided film on a long tape by a reel-to-reel process. Doubtlessly, CCs with double-sided configuration not only increase the materials utilization ratio, but also double the current carrying capacity and lead to higher engineering current density [6], [7]. However, the reports on double-sided CCs produced via solution method are limited in number [8]–[10]. Compared to vacuum thin film deposition techniques, the CSD method is an ex-situ route consisting of three steps: coating the substrate with the precursor solution, followed by pyrolysis and crystallization heat treatments. During the reel-to-reel processing, particular attention has to be paid to minimize the negative effects resulting from contact between the intermediate products (e.g. coated or pyrolyzed films) and the wheels. Especially, how to handle the wet film after coating is an important issue when designing the coating and heat treatment setup in order to obtain double-side coated tapes with uniform microstructure and properties.

In this paper, we proposed and described a reel-to-reel system, which combines a vertical dip coating and baking units. CeO_2 films simultaneously coated on both sides of one meter long home-made textured Ni-5at.%W tape was successfully achieved. A homogeneous texture over the length was achieved on both sides of the tape, demonstrating the feasibility of the reel-to-reel design for fabricating long-length textured templates for CCs.

II. EXPERIMENT DETAILS

A. Design of the Reel-to-Reel Coating and Drying System

In order to obtain double-sided coating simultaneously, a vertical dip coating unit combined with a drying system is proposed. Fig. 1 shows the schematic of the reel-to-reel setup which consists of three main parts, a take-up (upper, **A**) and a pay-off boxes (lower, **B**) connected by a furnace **C** with a quartz tube (10). The system is airtight, allowing coating and drying processes being performed under a controlled atmosphere. The withdrawing process is driven by a wheel (7) connected to a variable-speed motor controller. After vertically withdrawing from the precursor solution (2), the double-side

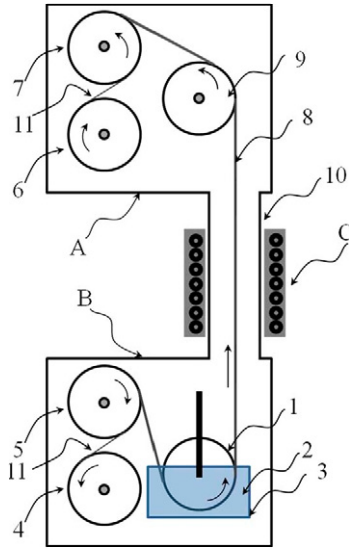


Fig. 1. Schematic of the reel-to-reel setup for coating and drying, where Teflon wheels 1, 4, 5, 6, 7, and 9 are fixed on the wall of the boxes, a precursor solution 2 is in a Teflon container 3, tape 8 and 11 are a coated metallic tape and plastic isolation, respectively.

coated wet tape was pre-heated. Eventually, the tape with dried film is wound up along with a thin KAPTON isolating tape by the take-up wheel in the upper box. There are two main achievements of this design. First, the contact between the coated wet film and the wheel is avoided. The dried film with double-sided coating can be handled easily afterwards, e.g. the guiding wheel (9) does not damage the intermediate products. Secondly, since further heat treatment for the tape is performed in another furnace tube, the withdrawal speed for coating (determining the film thickness) and the tape moving speed for crystallization (i.e. dwell time) can be controlled independently. Even though the drying process in the setup is linked with the dip coating, our preliminary results reveal that the dependence of drying procedures on epitaxial growth of CeO_2 thin films on NiW substrates is very limited as long as the drying temperature is optimized. Therefore, the design allows for a flexible coating condition for acquiring a double-sided film with desirable thickness, which could also be applied for other oxide thin films deposition.

B. Fabrication and Characterization of the CeO_2/NiW Template

Home-made one meter long biaxially textured Ni-5 at.% W tapes with $80\ \mu\text{m}$ thickness, 15 mm width were used as substrates. The preparation and properties of the NiW tapes are described elsewhere [11], [12].

CeO_2 films were produced by propionic acid based metal organic deposition (MOD) route, as reported previously [13]. The total cation concentration of the CeO_2 precursor solution and the withdrawal speed for coating were fixed at $0.3\ \text{mol L}^{-1}$, and $20\ \text{mm min}^{-1}$, respectively. After vertically withdrawing from the solution, the tape was continuously dried at 200°C in the furnace with a heating zone of 10 cm length. Both the coating and drying processes were performed under air atmosphere. Subsequently, the tape with dried film was crystallized at 1010°C in a furnace tube with flowing forming gas ($\text{Ar} + 5\% \text{H}_2$) in a continuous manner. The dwell time was about

30 min and controlled by the tape moving speed. The film thickness on each side is about 20 nm as determined by ellipsometry (FILMETRICS) measurements.

Phase and global texture of the tape at various positions over its length were examined by means of X-ray diffraction (XRD) with $\text{Cu K}\alpha$ radiation in a four-circle diffractometer (Bruker D8). The reflections of NiW (111), (002) and CeO_2 (111), (002) were used to characterize in-plane and out-of-plane texture, which is determined by the respective full width at half maximum (FWHM) values. Local surface texture analysis was carried out by electron backscattering diffraction (EBSD) using a scanning electron microscope (SEM, from Zeiss Supra 35) equipped with a HKL detector. The Kikuchi patterns were automatically analysed by the data handling software package (Channel 5). The microstructure of the tape was observed in the same microscope with an in-lens detector for high resolution images without coating any conductive layer.

To evaluate the quality of the CeO_2/NiW template, an all chemical solution derived YBCO/ $\text{Ce}_{0.9}\text{La}_{0.1}\text{O}_2/\text{Gd}_2\text{Zr}_2\text{O}_7/\text{CeO}_2$ structure was obtained on a short sample cut from the long piece prepared by reel-to-reel process. The $\text{Ce}_{0.9}\text{La}_{0.1}\text{O}_2$ (cap layer) and $\text{Gd}_2\text{Zr}_2\text{O}_7$ (barrier layer) were prepared by propionic acid based MOD route [14], [15], while the YBCO superconducting layer was prepared by low trifluoroacetate (Low TFA) MOD method [16]. The superconducting critical temperature T_c was determined in a cryogenic free measurement system (Cryogenic Ltd.).

III. RESULTS AND DISCUSSION

A. Texture and Morphology Study on Each Side of the CeO_2/NiW Template

$\theta - 2\theta$ XRD scans show that both sides of the tape form a CeO_2 phase with a strong c-axis orientation. Additionally, ϕ scans and ω scans on the substrate and the film were performed and the results are shown in Fig. 2. The FWHM values of the peaks determined by Gaussian fitting are also listed in Table I. The epitaxial relationship between the substrate and the film can be described as $\text{NiW (002)}\langle 001 \rangle // \text{CeO}_2(002)\langle 110 \rangle$, i.e. the CeO_2 lattice is in-plane 45° rotated with respect to the underlying NiW lattice. The similar FWHM values of the substrate and each side of the CeO_2 films reveal that the strong cube texture formed initially is transferred to the CeO_2 layer. The additional drying process at 200°C in air atmosphere has little effect on thin film growth. Comparable texture qualities on each side of the tape imply negligible influence of the contact between the coated film (side I) and the guiding wheel (9).

SEM micrographs of the CeO_2 films on each side of the tape are shown in Fig. 3. Both sides of the CeO_2 films are smooth, continuous and crack-free. The EBSD technique was also used to evaluate the surface texture and the grain boundary misorientation, as shown in Fig. 4. The grain boundary misorientation distributions on each side exhibit similar features, i.e., a large amount of the grains are connected with misorientation angles smaller than 10° . The two peaks in the distribution curves originate from two types of CeO_2 grain boundaries. The peak at about 1.5° corresponds to CeO_2 grains with small misorientation grown on individual NiW grains, while the other

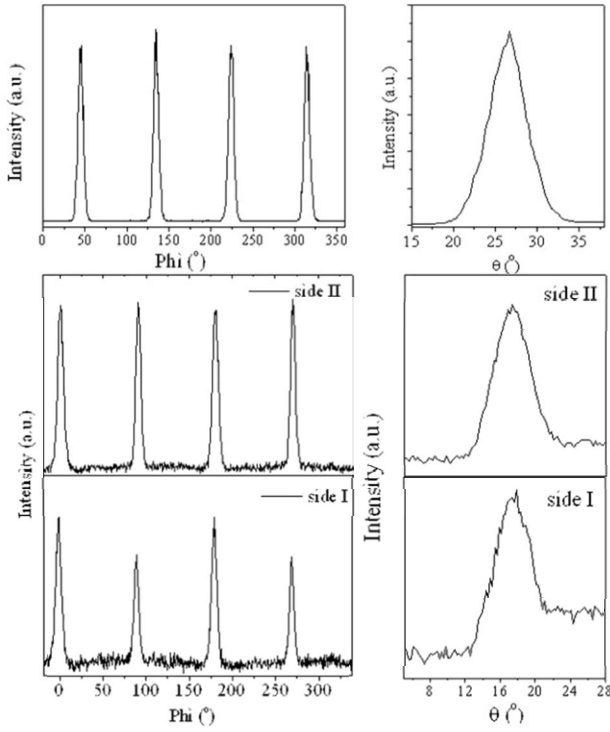


Fig. 2. ϕ scans (left) and ω scans (right) on the substrate (upper panel) and each side of the film (middle and lower panel).

TABLE I
QUANTITATIVE IN PLANE AND OUT PLANE TEXTURE ANALYSIS
OF EACH LAYER AND THE SUBSTRATE IN A SHORT
PIECE CUT FROM THE LONG TAPE

Layers	FWHM of rocking curve along RD ($^{\circ}$)	FWHM of rocking curve along TD ($^{\circ}$)	FWHM of phi ($^{\circ}$)
NiW	7.4	5.4	7.0
CeO ₂ (I)	7.0	5.7	6.7
CeO ₂ (II)	7.0	5.5	7.2

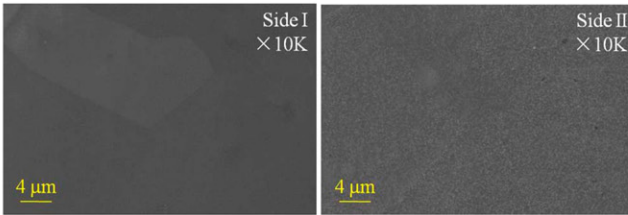


Fig. 3. SEM images of the CeO₂ films on each side of the tape.

peak at about 6° corresponds to those with larger misorientation inherited from the NiW grains boundaries. The four-fold concentrated pole figure acquired from an area of $500 \times 400 \mu\text{m}$ is indicative of high texture on the film surface in agreement with the global texture analysis.

The FWHM values of in-plane and out-of-plane texture on the NiW substrate and the CeO₂ (side II) were measured along the dip coating direction and plotted in Fig. 5. A small fluctuation is observed on the in-plane and out-of-plane orientation of both the NiW tape and the CeO₂ layer, but there is no clear correlation of the texture variation between the substrate and the film. Small standard deviation (S.d.) values are an indication of texture homogeneity over the tape length.

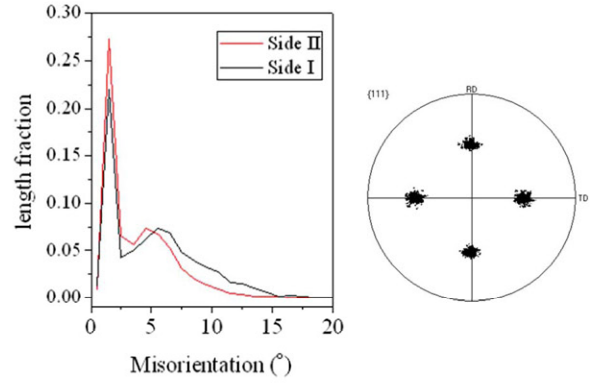


Fig. 4. EBSD analysis of Grain boundary misorientation distributions on each side of the tape (left), pole figure of CeO₂(111) (right).

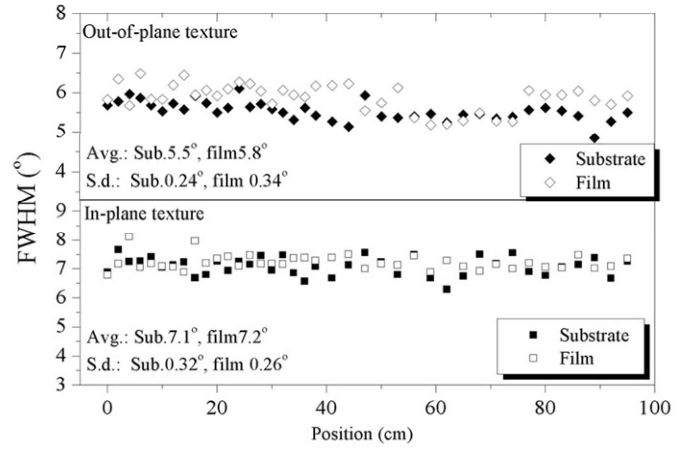


Fig. 5. FWHM values of ω scans (upper) and ϕ scans (lower) of both CeO₂ and NiW along the length of the dip-coated substrate.

B. Superconducting Test Structure

A full buffer layer architecture Ce_{0.9}La_{0.1}O₂/Gd₂Zr₂O₇/CeO₂ was prepared by CSD method. A 150 nm thick YBCO superconducting film on the buffered substrate was deposited using a low TFA solution MOD method in order to test the quality of the all chemical solution derived template. The YBCO film was coated by spin-coating technique. The reduced amount of TFA in the process is beneficial for shortening the pyrolysis time, while retaining a similar nucleation and growth mechanism of the YBCO films compared with the typical all TFA solution. In order to minimize the reactions between the template and the YBCO layer during the firing stage, a prolonged dwell time at a temperature as low as 740 $^{\circ}\text{C}$ was applied. A detailed study of YBCO layer deposition by using a reduced TFA solution will be described elsewhere [16]. The preliminary results show that the YBCO film forms a strong c-axis texture with a small weak (103) peak, even though weak NiWO₄ and BaCeO₃ peaks can be discerned. A T_c onset of 88 K was determined by AC susceptibility measurement. Further optimization of the YBCO deposition is ongoing.

IV. CONCLUSION

In this work, we demonstrated a reel-to-reel design for simultaneously depositing oxide films on textured technical substrates by conventional chemical solution method. One meter long highly textured double-sided CeO₂/home-made

Ni-5at.%W template was successfully obtained. The CeO₂ layers on both sides of the tape exhibit a high degree of biaxial texture confirmed by XRD and EBSD techniques. A homogenous microstructure with dense and crack free morphology is also observed. Texture homogeneity study over the whole length shows small variation of the FWHM value of in-plane and out-of-plane orientations, being indicative of the high quality of epitaxial growth realized in the continuous process. Finally, an all chemical solution derived YBCO/Ce_{0.9}La_{0.1}O₂/Gd₂Zr₂O₇/CeO₂ structure on a short sample resulted in an onset T_c of 88 K, demonstrating the possibility of producing long-length textured templates for coated conductors by a low cost route.

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